Advanced Measurement Capabilities

The special role that NIST plays in measurement metrology and standards often results in the development of instruments and facilities, as well as measurement methods, capable of probing material characteristics in ways unavailable elsewhere. These capabilities often provide the context and observations necessary to the development of a fundamental understanding of the mechanisms of material behavior. The Ceramics Division is privileged to have an excellent staff and world leaders who are responsible for the design and development of many of these unique instruments and facilities.

Debra L. Kaiser

Nanometrology

Working with instrument makers, the Nanotribology Group, directed by Dr. Stephen Hsu, has designed and built a remarkable facility featuring a cluster of equipment and instrumentation that establishes a new state of the art for conducting measurements and observations of nanoscale events. Included in this facility are a nanoadhesion apparatus, a dual white-light interferometer microscope, a multiscale friction tester, and a multifunction apparatus combining an ultrahigh vacuum scanning tunneling microscope with an atomic force microscope (UHV STM/AFM). Together, these instruments allow us to image, manipulate, and measure adhesion, friction, and other nanomechanical properties of materials from microscale to nanoscale dimensions.

Nanoadhesion Apparatus

Adhesion is an important issue in nanotechnology as well as in device manufacturing industries. Conventional adhesion measurement relies on large interfacial areas and the accurate positioning of two surfaces. Force measurements typically are performed by strain gauges and transducers with force sensitivities in the newton and millinewton ranges. These

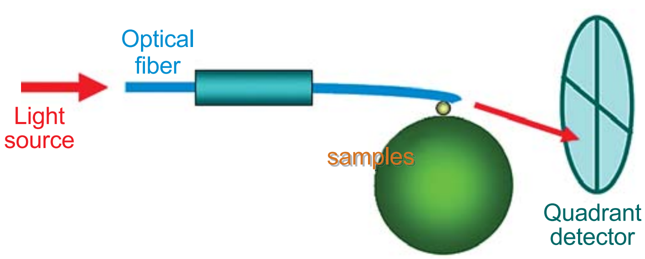

Figure 1: *Schematic operation of the NIST nanoadhesion apparatus.*

conventional instruments are unsuitable for nanoscale contacts. To resolve this issue, the newly designed NIST apparatus, Figure 1, allows adhesion force measurements between two surfaces at the nanoscale level using the AFM force measurement principle. In this device, capable of nanonewton force resolution, a laser light is piped through a fiber-optic cable directed at a quadrant photodetector. Because the length and stiffness of the fiber can be modified easily, a wide range of spring constants can be obtained, and the measurement of adhesion forces can be achieved at multiple force levels. Thus, the NIST apparatus has established an exciting new opportunity to measure the influence of surface forces on adhesion, molecular interactions, and the compliance of surfaces, all critical data to device manufacturing industries.

UHV STM/AFM

Enhanced by the superior vibration isolation and clean room environment in NIST's new Advanced Measurement Laboratory (AML), our UHV STM/AFM system, Figure 2, provides atomic imaging and force measurement with an unprecedented resolution and accuracy.

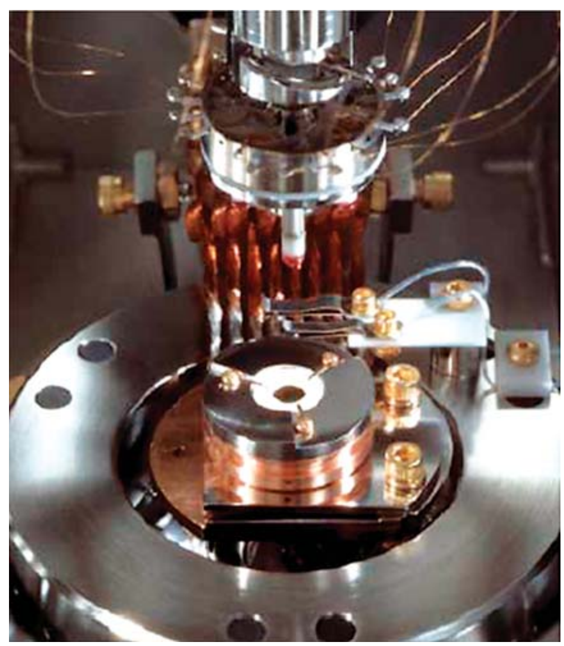

Figure 2: *Ultrahigh vacuum STM/AFM apparatus.*

Combined with our conventional AFM, equipped with a "triboscope" attachment, we are able to image and manipulate surface features and measure a wide range of material characteristics and properties important in nanodevice operation. As a result, we now are leading fundamental metrological efforts, working with device and magnetic hard disk industries, as well as academic institutions, to establish reliable measurement methods, calibration artifacts, and infrastructural support for current and future industries.

Multiscale Friction Tester

At the nanoscale level, friction between movable components is a significant concern. To address this issue, NIST and Hysitron, Inc. have collaborated to develop a three-dimensional, capacitance based, force sensor. The stiffness and position of the probe tips can be tightly controlled to yield accurate information of the contact area. Force measurements can be made in the range from 100 nN to 1 N in the z-direction, and 500 nN to 0.5 N in the x,y-directions. Displacements can be measured from 0.2 nm to 5 µm in the z-direction, and 10 nm to 15 µm in the x,y-directions. Consequently, accurate friction measurements can be conducted at length scales ranging from the nanometer scale to the micrometer scale. Already, this instrument has enabled us to resolve apparent disparities in nanofriction measurements and to understand the nature of what had been thought to be anomalous friction behavior. (See Friction Scaling in the Technical Highlights section of this report.)

High Resolution X-ray Metrology

Powder and single crystal x-ray diffraction are widely used in industry, research facilities, and academia as one of the principal means of characterizing materials. Both techniques yield a wealth of information on the crystallographic and microstructural character of the specimen. The powder diffraction method has the virtue that it can probe a continuous sequence of crystallographic reflections with a single scan in angular space, while single crystal methods can be adapted to high resolution analysis of materials such as thin films.

However, results from both powder and high-resolution techniques are affected by a complex optical aberration function that is specific to the diffraction optics and goniometer assembly used in the experiment. NIST Standard Reference Materials (SRMs) are the recognized means by which these aberrations may be characterized to achieve improved measurement accuracy. To address these issues, Dr. James Cline of the Data and Standards Technology Group conceived and designed the Ceramics Division Parallel Beam Diffractometer (CDPBD).

The CDPBD, Figure 3, was designed and built specifically to perform traceable measurements on powder and thin film specimens. Installed in the new NIST Advanced Measurement Laboratory, this facility provides the environmental and temperature controls requisite for a new generation of NIST SRMs that will enable unprecedented measurement accuracy in a highly competitive, data conscious, materials research community.

Figure 3: *The Ceramics Division Parallel Beam Diffractometer in the AML.*

X-ray Absorption Spectroscopy

Advances in our x-ray absorption spectroscopy facilities have achieved an unrivaled capability enabling our Characterization Methods Group (CMG) to address a remarkably broad range of challenging structure and chemistry issues at the forefront of materials science research today. Through the application of a truly unique combination of beamline facilities, we are able to examine characteristics of surfaces, interfaces, and bulk materials in a manner heretofore inaccessible.

To achieve this capability, CMG, led by Dr. Daniel Fischer, brought together a suite of three unique high-throughput x-ray spectroscopy beamlines (designated U7A, X24A, and X23A2). Housed in the National Synchrotron Light Source located at Brookhaven National Laboratory in New York, these beamlines, taken together, can easily examine nearly all of the naturally occurring elements in the entire periodic table. This year, the capabilities of beamline U7A, used for soft x-ray materials science applications, were significantly enhanced by the addition of a 14 element, state-of-the-art, Si (Li) fluorescence yield detector, providing best in the world resolution. Further enhancement of U7A, Figure 4, was achieved through the installation of a 6-axis manipulator that enables the exciting prospect of molecular alignment studies.

Figure 4: The soft x-ray spectroscopy station on beamline U7A.

Building on these capabilities, CMG has initiated a long-term plan, co-funded with Sandia National Laboratory, for establishing a variable energy XPS (x-ray photoelectron spectroscopy) and NEXAFS/EXAFS (near-edge/extended x-ray absorption fine structure) scientific program utilizing beamline X24A. A new, fully automated materials science end-station is planned, modeled after the very successful high throughput attained on U7A. The emphasis for this work will be on the use of variable energy XPS for chemical depth profiling, sub-surface chemistry, and interface chemistry.

X-ray Topography

The premier, dedicated, monochromatic topography facility in the U.S. has been developed and implemented at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL), Argonne, Illinois. X-ray topography is used to study the defect microstructure of single-crystal materials by imaging the diffracted intensity from selected lattice planes of the sample. The NIST effort, led by Dr. David Black, takes advantage of the third-generation synchrotron x-ray source to provide a spatially enlarged beam, up to 140 mm x 50 mm, with large-area real-time imaging detectors to perform static and dynamic experiments with large samples. In addition to topography, radiographic and phase contrast imaging are available. This instrument has recently been used to study subsurface damage in 300 mm production silicon wafers, defects in thin strained-silicon films, strain around nanoindentations and dendrite growth in Sn/Bi alloys. In the case of the strained-silicon films, by using grazing incidence geometry we have been able to directly image defects in a film ≈ 165 Å thick. These defects have been shown to mimic the misfit dislocation structure in the underlying relaxed Si/Ge virtual substrate. Understanding how the substrate defects propagate into the strained-silicon film is a critical issue in the development of the next generation of strained silicon electronic devices.

Ultrasmall-Angle X-ray Scattering

Our ultrasmall-angle x-ray scattering (USAXS) instrument, constructed as part of the UNICAT facility, is housed at the APS at ANL. The NIST effort is led by Dr. Andrew Allen. The facility provides the most versatile SAXS facility for materials science and engineering research in the world. Using a single instrument configuration, the representative microstructures within a wide range of heterogeneous materials of technological importance can be quantified over a scale range extending from nanometers to micrometers. The continuously tunable x-ray energy enables anomalous SAXS studies to be made, allowing different microstructural components within a composite system to be distinguished. Microstructures also can be measured when they possess an arbitrarily high anisotropy, such as those encountered in some coating materials and membranes. Recently, the high APS brilliance has been combined with the instrument's high-precision x-ray crystal optics to provide a spatial resolution on the order of 10 µm for resolving the microstructural gradients within solid oxide fuel cell (SOFC) components.

The USAXS instrument serves microstructure metrology needs in a wide class of technological problems encountered in metals and alloys, structural ceramics, thermal barrier coatings, fuel cells, biological scaffolds, flowing nanoparticle suspensions, soot in flames, and polymer gels. Furthermore, the intrinsic absolute intensity calibration provided by this instrument underpins our partnerships with others to develop specialized SAXS instrumentation for interrogating surface structures in electronic gate materials, or to approach sub-micrometer spatial resolution in SOFC research.

For More Information on this Topic

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